Functional Guidelines for Evaluating Organic Analysis

CASE No.: 40200 **SDG No.: B0003, B0005**
LABORATORY: A4 **SITE: Riverside Ave**
SAMPLER: TTEMI **ANALYSIS: PEST**

DATA ASSESSMENT

The current SOP HW-36 (Revision 1) August 2007, USEPA Region II Data Validation SOP for Statement of Work SOM01.2 for evaluating organic data have been applied.

Data has been reviewed according to TDF specifications, the National Functional Guidelines Report and the CCS Semi- Automated Screening Results Report.

All data are valid and acceptable except those analytes rejected "R"(unusable). Due to the detection of QC problems, some analytes may have the "J" (estimated), "N" (presumptive evidence for the presence of the material), "U" (non-detect) or "JN"(presumptive evidence for the presence of the material at an estimated value) flag. All action is detailed on the attached sheets.

The "R" flag means that the associated value is unusable. In other words, significant data bias is evident and the reported analyte concentration is unreliable.

Reviewer's Signature: **Dorina Christina Alliu** **Date: August/21/2010**

Peer Reviewer's Signature: _____ **Date: / /2010**

Verified By: _____ **Date: / /2010**

SDG# B0003

1. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detects (sample quantitation limits) will be flagged as estimated, "J", or unusable, "R", if the holding times are grossly exceeded.

The following action was taken in the samples and analytes shown due to excessive holding time.

The following pesticide water and/or soil samples are outside primary analysis holding time criteria. Detected compounds are qualified J. Non-detected compounds are qualified UJ.

B0010

2. SURROGATES

All samples are spiked with surrogate compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below.

No problems found for this qualification

3. MATRIX SPIKE/SPIKE DUPLICATE, MS/MSD:

The MS/MSD data are generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD may be used in conjunction with other QC criteria for additional qualification of data.

Not applicable

4. LABORATORY CONTROL RECOVERY (LCS):

The LCS data is generated to determine the long-term precision and accuracy of the analytical method. The LCS may be used in conjunction with other QC criteria for additional qualification of data.

No problems found for this qualification

5. BLANK CONTAMINATION:

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Depending on the concentration of the analyte in the blank, the analytes are qualified as non-detects, "U".

The following analytes in the sample shown were qualified "U" for these reasons:

A) Method/Instrument blank contamination:

No problems found for this qualification

B) Field or rinse blank contamination:

No problems found for this qualification

6. CALIBRATION:

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.

A) Percent Relative Standard Deviation (%RSD) and Percent Difference (%D):

For the PESTICIDE fraction, if %RSD exceeds 20% for all analytes except alpha-BHC and delta-BHC 25%, for the two surrogates and Toxaphene 30%, qualify all associated positive results "J" and non-detects are not qualified.

B) The Percent Difference (%D) for each of the SCP and surrogate in the PEM used for CCV must be greater than or equal to-25% and less than or equal to 25.0%. The Percent Difference (%D) between the calibration Factor (CF) for each of the SCP and surrogate in the Calibration Verification Standard (CS3) and the mean calibration factor from the initial calibration must be greater than or equal to-20% and less than or equal to 20.0%. The Percent Difference not within limits, detected associated compounds are qualified "J" and non-detected associated compounds are qualified "UJ".

The following analytes in the sample shown were qualified for %RSD and %D:

The following pesticide samples are associated with % RSD exceeding criteria Detected compounds are qualified J. Non-detected compounds are not qualified.

Methoxychlor B0003, B0004, B0010, B0012, B0031, PBLBQK, PLCSQK

The following pesticide samples are associated with the percent resolution that did not meet the resolution criteria. Detected compounds are qualified JN. Non-detected compounds are qualified R.

44DDD B0003, B0004, B0010, B0012, B0031, PBLBQK, PLCSQK

The following pesticide samples are associated with a continuing PEM in which the percent difference between the nominal and calculated amounts for a PEM compounds is outside criteria. Detected compounds are qualified J. Non-detected compounds are qualified UJ.

Tetrachloro-m-xylene B0003, B0004, B0012, B0031, PBLKQK

4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Aldrin, Dieldrin, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin aldehyde, Endrin ketone, Heptachlor, Heptachlor epoxide, Methoxychlor, Toxaphene, alpha-BHC, alpha-Chlordane, beta-BHC, delta-BHC, gamma-BHC (Lindane), gamma-Chlordane

7. COMPOUND IDENTIFICATION:

The retention times of reported compounds must fall within the calculated retention time windows for the two chromatographic columns and a GC/MS confirmation is required if the concentration exceeds 10ng/ml in the final sample extract.

The following pesticide samples have percent differences between analyte results in the range of 26-50%. Detected compounds are qualified J.

Heptachlor epoxide PLCSQK

Gamma-BHC (Lindane) PLCSQK

4,4'-DDE PLCSQK

The following pesticide samples have percent differences between analyte results in the range of 51-100%. Detected compounds are qualified NJ.

alpha-BHC B0010

8. CONTRACT PROBLEMS NON-COMPLIANCE: No problems.

9. FIELD DOCUMENTATION: No problems.

10. OTHER PROBLEMS:

None

11. This package contains re- extractions, re-analyses or dilution runs. Upon reviewing the QA results, the following Form 1(s) are identified NOT to be used.

None

SDG# B0005

1. HOLDING TIME:

The amount of an analyte in a sample can change with time due to chemical instability, degradation, volatilization, etc. If the specified holding time is exceeded, the data may not be valid. Those analytes detected in the samples whose holding time has been exceeded will be qualified as estimated, "J". The non-detects (sample quantitation limits) will be flagged as estimated, "J", or unusable, "R", if the holding times are grossly exceeded.

The following action was taken in the samples and analytes shown due to excessive holding time.

The following pesticide water and/or soil samples are outside primary analysis holding time criteria. Detected compounds are qualified J. Non-detected compounds are qualified UJ.

B0005, B0006, B0009, B0013, B0014, B0015, B0016

2. SURROGATES

All samples are spiked with surrogate compounds prior to sample preparation to evaluate overall laboratory performance and efficiency of the analytical technique. If the measured surrogate concentrations were outside contract specifications, qualifications were applied to the samples and analytes as shown below.

The following pesticide samples have surrogate percent recoveries that are greater than 200% Detected compounds are qualified J. Non-detected compounds are not qualified.

Decachlorobiphenyl PLCSSI

4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Aldrin, Dieldrin, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin aldehyde, Endrin ketone, Heptachlor, Heptachlor epoxide, Methoxychlor, Toxaphene, alpha-BHC, alpha-Chlordane, beta-BHC, delta-BHC, gamma-BHC (Lindane), gamma-Chlordane

Tetrachloro-m-xylene B0005

The following pesticide samples have surrogate percent recoveries outside the lower limit of the criteria window, but greater than 10%. Detected compounds are qualified J. Non-detected compounds are qualified UJ.

Decachlorobiphenyl B0005

4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Aldrin, Dieldrin, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin aldehyde, Endrin ketone, Heptachlor, Heptachlor epoxide, Methoxychlor, Toxaphene, alpha-BHC, alpha-Chlordane, beta-BHC, delta-BHC, gamma-BHC (Lindane), gamma-Chlordane

The following pesticide method blanks have surrogate percent recoveries outside criteria. Detected compounds are qualified J. Non-detected compounds are not qualified.

Decachlorobiphenyl PBLKSI

4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Aldrin, Dieldrin, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin aldehyde, Endrin ketone, Heptachlor, Heptachlor epoxide, Methoxychlor, Toxaphene, alpha-BHC, alpha-Chlordane, beta-BHC, delta-BHC, gamma-BHC (Lindane), gamma-Chlordane

3. MATRIX SPIKE/SPIKE DUPLICATE, MS/MSD:

The MS/MSD data are generated to determine the long-term precision and accuracy of the analytical method in various matrices. The MS/MSD may be used in conjunction with other QC criteria for additional qualification of data.

Not Applicable

4. LABORATORY CONTROL RECOVERY (LCS):

The LCS data is generated to determine the long-term precision and accuracy of the analytical method. The LCS may be used in conjunction with other QC criteria for additional qualification of data.

The following pesticide samples are associated with a laboratory control sample (LCS) with percent recoveries outside the lower limit of the criteria window. Detected compounds are qualified J. Non-detected compounds are qualified R.

Endosulfan sulfate B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI

5. BLANK CONTAMINATION:

Quality assurance (QA) blanks, i.e., method, field, or rinse blanks are prepared to identify any contamination, which may have been introduced into the samples during sample preparation or field activity. Method blanks measure laboratory contamination. Field and rinse blanks measure cross-contamination of samples during field operations. Depending on the concentration of the analyte in the blank, the analytes are qualified as non-detects, "U".

The following analytes in the sample shown were qualified "U" for these reasons:

A) Method/Instrument blank contamination:

No problems found for this qualification

B) Field or rinse blank contamination:

No problems found for this qualification

6. CALIBRATION:

Satisfactory instrument calibration is established to ensure that the instrument is capable of producing acceptable quantitative data. An initial calibration demonstrates that the instrument is capable of giving acceptable performance at the beginning of an experimental sequence. The continuing calibration checks document that the instrument is giving satisfactory daily performance.

A) Percent Relative Standard Deviation (%RSD) and Percent Difference (%D):

For the PESTICIDE fraction, if %RSD exceeds 20% for all analytes except alpha-BHC and delta-BHC 25%, for the two surrogates and Toxaphene 30%, qualify all associated positive results "J" and non-detects are not qualified.

B) The Percent Difference (%D) for each of the SCP and surrogate in the PEM used for CCV must be greater than or equal to-25% and less than or equal to 25.0%. The Percent Difference (%D) between the calibration Factor (CF) for each of the SCP and surrogate in the Calibration Verification Standard (CS3) and the mean calibration factor from the initial calibration must be greater than or equal to-20% and less than or equal to 20.0%. The Percent Difference not within limits, detected associated compounds are qualified "J" and non-detected associated compounds are qualified "UJ".

The following analytes in the sample shown were qualified for %RSD and %D:

The following pesticide samples are associated with % RSD exceeding criteria Detected compounds are qualified J. Non-detected compounds are not qualified.

Methoxychlor B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI, PLCSSI

The following pesticide samples are associated with a continuing PEM in which the percent difference between the nominal and calculated amounts for a PEM compounds is outside criteria. Detected compounds are qualified J. Non-detected compounds are qualified UJ.

Decachlorobiphenyl B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI

4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Aldrin, Dieldrin, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin aldehyde, Endrin ketone, Heptachlor, Heptachlor epoxide, Methoxychlor, Toxaphene, alpha-BHC, alpha-Chlordane, beta-BHC, delta-BHC, gamma-BHC (Lindane), gamma-Chlordane

alpha-BHC B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI

4,4'-DDT B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI

Gamma-BHC (Lindane) B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI

Endrin B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI

Methoxychlor B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI

Tetrachloro-m-xylene B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI

4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Aldrin, Dieldrin, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin aldehyde, Endrin ketone, Heptachlor, Heptachlor epoxide, Methoxychlor, Toxaphene, alpha-BHC, alpha-Chlordane, beta-BHC, delta-BHC, gamma-BHC (Lindane), gamma-Chlordane

The following pesticide samples are associated with a CCV with % Difference exceeding criteria Detected compounds are qualified J. Non-detected compounds are qualified UJ.
B0005, B0006, B0009, B0013, B0014, B0015, B0016

Decachlorobiphenyl B0005, B0006, B0009, B0013, B0014, B0015, B0016, PLCSSI

4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Aldrin, Dieldrin, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin aldehyde, Endrin ketone, Heptachlor, Heptachlor epoxide, Methoxychlor, Toxaphene, alpha-BHC, alpha-Chlordane, beta-BHC, delta-BHC, gamma-BHC (Lindane), gamma-Chlordane

alpha-BHC B0005, B0006, B0009, B0013, B0014, B0015, B0016

delta-BHC B0005, B0006, B0009, B0013, B0014, B0015, B0016

Endrin ketone B0005, B0006, B0009, B0013, B0014, B0015, B0016

Gamma-BHC (Lindane) B0005, B0006, B0009, B0013, B0014, B0015, B0016

Dieldrin B0005, B0006, B0009, B0013, B0014, B0015, B0016

Endrin B0005, B0006, B0009, B0013, B0014, B0015, B0016

Methoxychlor B0005, B0006, B0009, B0013, B0014, B0015, B0016

4,4'-DDD B0005, B0006, B0009, B0013, B0014, B0015, B0016

Endrin aldehyde B0005, B0006, B0009, B0013, B0014, B0015, B0016

Tetrachloro-m-xylene B0005, B0006, B0009, B0013, B0014, B0015, B0016

4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Aldrin, Dieldrin, Endosulfan I, Endosulfan II, Endosulfan sulfate, Endrin, Endrin aldehyde, Endrin ketone, Heptachlor, Heptachlor epoxide, Methoxychlor, Toxaphene, alpha-BHC, alpha-Chlordane, beta-BHC, delta-BHC, gamma-BHC (Lindane), gamma-Chlordane

Endosulfan I B0005, B0006, B0009, B0013, B0014, B0015, B0016

4,4'-DDE B0005, B0006, B0009, B0013, B0014, B0015, B0016

4,4'-DDT B0005, B0006, B0009, B0013, B0014, B0015, B0016

Heptachlor B0005, B0006, B0009, B0013, B0014, B0015, B0016

The following pesticide samples are associated with the percent resolution that did not meet the resolution criteria. Detected compounds are qualified JN. Non-detected compounds are qualified R.

4,4'-DDD B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI

Endosulfan I B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI

The following pesticide samples are associated with Combined % Breakdown exceeding criteria.

If Endrin is detected then: Detects for Endrin are qualified J. Detects for Endrin aldehyde and Endrin ketone are qualified J.

If Endrin is not detected then: Detects for Endrin are qualified R. Detects for Endrin aldehyde and Endrin ketone are qualified JN.

If DDT is detected then: Detects for 4,4' DDT are qualified J. Detects for 4,4' DDD and 4,4' DDE are qualified J.

If DDT is not detected then: Detects for 4,4' DDT are qualified R. Detects for 4,4' DDD and 4,4' DDE are qualified JN.

B0005, B0006, B0009, B0013, B0014, B0015, B0016, PBLKSI, PLCSSI

7. COMPOUND IDENTIFICATION:

The retention times of reported compounds must fall within the calculated retention time windows for the two chromatographic columns and a GC/MS confirmation is required if the concentration exceeds 10ng/ml in the final sample extract.

The following pesticide samples have percent differences between analyte results exceeding 50% and the results are below CRQL. Using professional judgment Detected compounds are qualified J.

Gamma-BHC (Lindane) PLCSSI

Dieldrin PLCSSI

Endrin PLCSSI

4,4'-DDE PLCSSI

8. CONTRACT PROBLEMS NON-COMPLIANCE: No problems.

9. FIELD DOCUMENTATION: No problems.

10. OTHER PROBLEMS:

None

11. This package contains re- extractions, re-analyses or dilution runs. Upon reviewing the QA results, the following Form 1(s) are identified NOT to be used.

None